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# Combustibility Tests of 1,1,1,2-tetrafluoroethane in a Simulated Compressor Cylinder

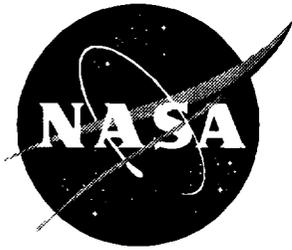
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June 1997





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## Contents

1.0 Introduction .....	1
2.0 Equipment Description .....	2
2.1 Test Vessel .....	2
2.2 Ignitors .....	2
2.3 Instrumentation and Data Acquisition .....	3
2.4 Charge-and-Purge System .....	3
3.0 Results .....	3
3.1 Exploding-Wire Ignitor .....	3
3.1.1 R134a-Air .....	4
3.1.2 R134a-Air-N <sub>2</sub> .....	6
3.2 Cartridge-Heater Tests .....	7
3.3 Flame Propagation Through an Obstruction .....	8
3.4 Autoignition Tests .....	8
3.5 Decomposition Tests .....	8
4.0 Concluding Remarks .....	9
5.0 Reference .....	9



## Abstract

*The advantages of high-molecular-weight gas (heavy gas) as a wind-tunnel medium have been recognized for some time. The current heavy gas of choice chlorofluorocarbon-12 (CFC-12) (refrigerant R12) for the Transonic Dynamics Tunnel (TDT) must be replaced because manufacture of this gas ceased in 1995. An attractive replacement is 1,1,1,2-tetrafluoroethane (refrigerant R134a). Acceptable properties of this gas include molecular weight and speed of sound. Its vapor pressure allows simplified reclamation from mixtures with air. However, it is recognized that R134a is combustible under certain conditions of temperature, pressure, and concentration. A comprehensive study was conducted to identify those conditions and the influence of various parameters on the combustibility of the gas-air mixture.*

## 1.0 Introduction

The Transonic Dynamics Tunnel (TDT) is a large-scale wind tunnel at the Langley Research Center. The TDT uses a high-molecular-weight gas (heavy gas) for improved testing characteristics, such as increased Reynolds number, lower speed of sound, and lower power requirements. Currently, the tunnel uses chlorofluorocarbon-12 (CFC-12, refrigerant R12), production of which ceased December 1995, as the test medium. A major project is under way to convert the heavy-gas medium from R12, to hydrofluorocarbon-134a (HFC-134a, refrigerant R134a). However R134a is combustible under certain conditions. Properties of HFC-134a are given in table 1.

The TDT is a continuous-flow, closed-loop, subatmospheric-pressure tunnel with a 16-ft<sup>2</sup> test section and an approximately 1 000 000-ft<sup>3</sup> internal volume. The exchange of air to heavy gas is made by pumping the tunnel to low pressure then vaporizing liquid R134a and injecting it into the bottom of the tunnel while extracting air from the top. Once a purity of about 95 percent molar concentration of R134a in air is achieved, no additional gas is drawn from the top. Heavy-gas injection continues until the desired test pressure is achieved. This process is reversed for the exchange of heavy gas to air to permit tunnel access. The heavy-gas-air mixture leaving the tunnel and entering the heavy-gas-reclamation system may range from zero to nearly 100 percent R134a. The reclamation system separates the heavy gas from the air by compressing and cooling the gas-air mixture, thereby condensing the heavy gas. The liquid R134a is then returned to the storage tank for future use. Compression of the gas is accomplished by a five-stage, double-acting, reciprocating compressor, with intercooling at each stage. During this compression and cooling process, the gaseous mixture is subjected to a wide range of pressures, temperatures, and molar concentrations.

An internal study of the reclamation system identified the compressor as the only viable ignition source in the system. Therefore, a comprehensive test program was

initiated to determine the combustibility envelope of R134a and the peak pressure resulting from combustion. The test apparatus diameter, length, and material were chosen to simulate the upper-stage cylinder of the compressor. Typically, testing was conducted at an initial temperature of 350°F because this temperature corresponds to the highest expected gas temperature based upon an adiabatic compression process in the cylinder of the compressor during normal operation.

The first series of tests determined the combustibility envelope of R134a-air with an American Society for Testing and Materials (ASTM) standard exploding-wire ignition source (ref. 1) and resultant peak pressure as a function of heavy-gas concentration. Additional tests were conducted to determine the effects of nitrogen diluent on the combustibility of the mixture. These tests investigated the feasibility of rendering gas mixtures in the TDT noncombustible.

The effect of a high-temperature surface in contact with the combustible mixture was investigated by means of a cartridge heater that extended into the chamber and heated the test vessel walls. It was discovered that, above some threshold temperature, the R134a-air mixture may spontaneously react without a concentrated ignition source. A series of tests were conducted to determine the effects of initial pressure and concentration on the temperature at which these reactions occur. This temperature is taken as the autoignition temperature of the mixture. The influence of small amounts of compressor oil (Tribol 890) on autoignition temperature was also investigated.

The phenomenon of flame propagation into the compressor downstream piping is of critical importance. A series of tests were conducted to determine if a flame could proceed through a compressor discharge valve into a large chamber of R134a-air mixture and propagate the length of the chamber.

This paper characterizes the combustibility envelope of R134a in the presence of air and oxygen-lean (less than 21 percent O<sub>2</sub> molar concentration) air for a standard ignitor, the spontaneous ignition of an

R134a-air mixture, and the ability of an ignited mixture to propagate through a restriction and into a pipe with cool walls.

## 2.0 Equipment Description

### 2.1 Test Vessel

The test vessel consisted of a section of ASTM A106, 6-in., schedule-80 pipe with ASTM A105, 900-lb class, slip-on flanges on each end. Penetrations were made in two 900-lb blind flanges for charging, venting, and instrumentation, including thermocouples, pressure transducers, and ignitors. The cylindrical inner volume had a 5.761-in. inside diameter (ID) and was 17 in. long for a total volume of 0.2564 ft<sup>3</sup> (7.26 L). Vessel temperature was maintained at fixed set points with four 2500-W band heaters on the flanges and two 1250-W band heaters on the pipe section. Temperature was controlled by a Watlow model 965 controller. Figure 1 shows the apparatus arrangement with thermocouple and heater location.

For flame-propagation studies, the apparatus was extended by adding a 6-ft-long section of ASTM A106, 6-in., schedule-80 pipe with a series of thermocouples along the centerline. Figure 2 shows the arrangement of the extended apparatus. Between the original apparatus and the extension is a discharge valve taken from the compressor. A schematic of the compressor discharge valve is shown in figure 3. The flanges of the extension are heated with 2500-W band heaters. The pipe extension was heated with five 1250-W band heaters.

### 2.2 Ignitors

Two ignition sources were used with the test apparatus. An exploding wire was provided by brazing copper

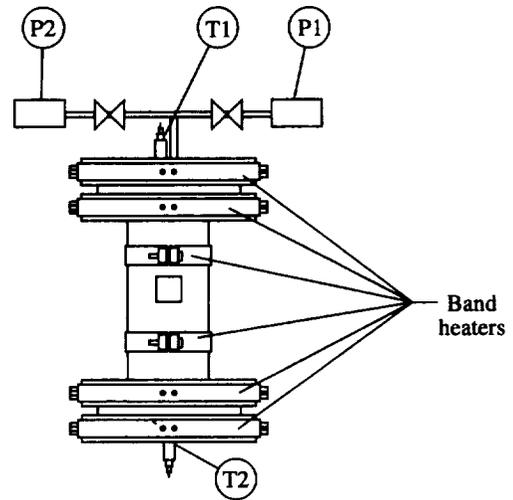


Figure 1. Test fixture for HFC-134A exploding-wire and autoignition tests.

extensions to the terminals of a nonresistor-type spark plug and soldering a 0.076-mm-diameter, tinned-copper wire about 0.5 in. long to the extensions. Energy to the ignitor was supplied from a 110-V alternating current (ac) source. This type of ignitor is an ASTM standard (E918-83) ignition source for combustibility testing (ref. 1).

The other ignition source consisted of a 150-W, 0.25-in.-diameter cartridge heater that was 11 in. long, with the last inch heated. Heater temperature was monitored by an internally mounted thermocouple located in the heated section. The cartridge heater was controlled by a proportional-integral-differential (PID) controller and typically was ramped to a fixed temperature and held for a fixed time. Autoignition tests were conducted by ramping the set point of the vessel wall temperature.

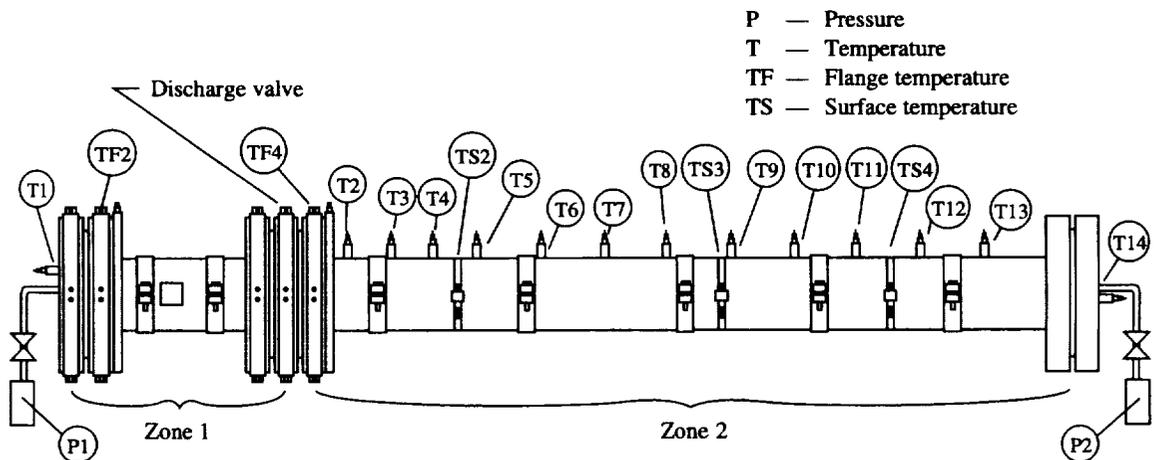


Figure 2. Test fixture for flame propagation studies, 7.5-ft-long x 5.76-in. ID carbon steel pipe.

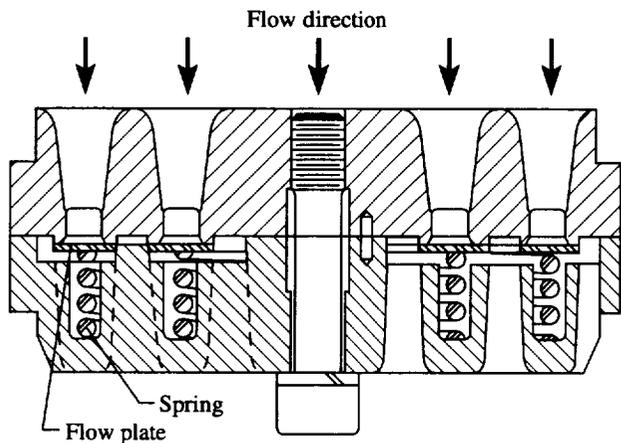


Figure 3. Compressor discharge valve.

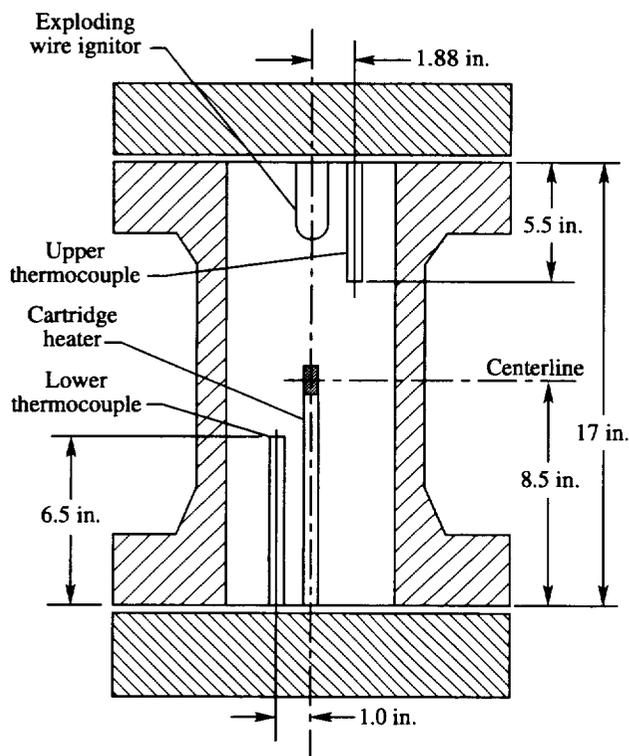


Figure 4. Short apparatus.

### 2.3 Instrumentation and Data Acquisition

Figure 4 shows the interior locations of the ignitors and thermocouples. All thermocouples were type J, iron-constantan, with 1/8-in. 304 stainless steel sheath. The thermocouples that measured flange temperatures were bayonet-mounted 1/8 in. from the inner surface of the flanges. A pressure transducer was mounted in each end of the apparatus. The chamber pressure transducer had a

range of 0–2000 psig and a transient response of 20 kHz, while the pipe extension end transducer had a range of 0–1500 psig and a transient response of 20 kHz. Accuracy for each instrument was 0.5-percent full-scale.

Data were recorded on a standard VHS tape with a Metrum data-acquisition recorder. This recorder stores up to 16 channels of input and can record at a 20-kHz rate. For these tests, a recording rate of 312 samples/sec/channel was used. From the Metrum, the data were downloaded and stored on disk. Plotting was performed on a personal computer. Plots were also made on a Soltech strip-chart recorder for immediate viewing.

### 2.4 Charge-and-Purge System

Figure 5 shows a schematic of the charge-and-purge system. The R134a was introduced into the vessel at known pressure followed by N<sub>2</sub>, if used, then bottled compressed “breathing” air at –70°F dew point. The desired pressures were determined beforehand to give a known partial pressure of each gas. Mixing was accomplished by introducing the gases through an offset high-velocity jet. Ten minutes were allowed to elapse after charging to facilitate mixing and stabilization. For the flame propagation studies, a mixing tank or “run” tank was used. The run tank had an approximately 10-ft<sup>3</sup> volume and thermostatically controlled heaters to maintain temperature. The run tank was initially evacuated then charged to a known partial pressure for each gas. The apparatus was evacuated to approximately 1 torr then charged to the desired total pressure with the contents of the run tank.

### 3.0 Results

Results of the combustibility tests are discussed below, and the tabulated data are presented in tables 1–6. Results are grouped by ignition source, mixture composition, and test.

#### 3.1 Exploding-Wire Ignitor

The exploding-wire ignitor tests were conducted with an ASTM standard ignition source, as described in section 2.2. The mechanism for providing ignition consists of passing a current through the thin (0.076-mm-diameter) copper wire. At a local site, the wire melts through, causing an arc that creates an extremely high temperature in a concentrated location. This ignition source is consistent and provides repeatable results. With this ignition source, R134a-air and R134a-air-nitrogen mixtures were tested. The tests with nitrogen added to the mixture were performed to determine the molar fraction needed to render the mixture noncombustible. Addition of nitrogen to the TDT reclamation system had been

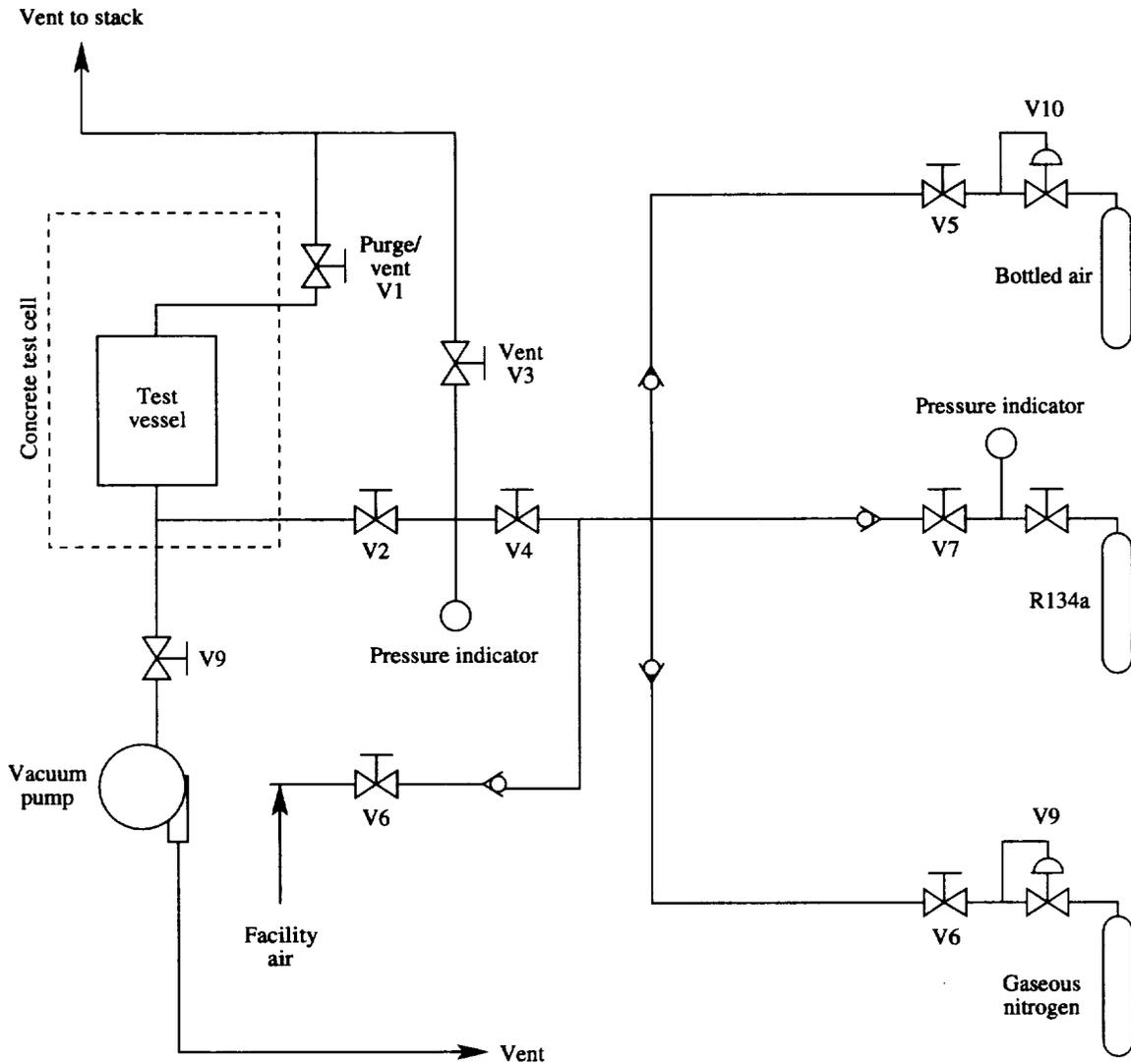


Figure 5. R134a charge-and-purge schematic.

considered as a means to mitigate the risk of mixture ignition. The test results from the exploding-wire ignition source appear in tables 2(a) and (b).

**3.1.1 R134a-Air.** The R134a-air exploding-wire combustion tests were conducted in the short-test apparatus described in section 2.0 and shown in figure 1. All tests were conducted with an initial temperature of 350°F, which is the highest expected adiabatic compression temperature of the compressor. Tests were conducted over a pressure range of 75–400 psig and molar concentrations ranging from 8–45 percent R134a. Combustion was defined as a 7-percent increase in pressure per ASTM E918-83 (ref. 1).

Results are tabulated in tables 2(a), (b), and (c) which list molar concentration, charge pressure for each gas, equivalence ratio, peak pressure, ratio of initial to

final pressure, and peak temperature indicated on the upper and lower gas thermocouples. Charge pressures are recorded as gauge pressure after the addition of each gas; thus, “R134a pressure (psig)” is the sum of the R134a added and the atmospheric pressure of air originally present in the test vessel. “N<sub>2</sub> pressure (psig)” is the sum of R134a, N<sub>2</sub>, and original air. “Air pressure” is the total gauge pressure of all the gases added to the vessel. These tests establish the combustibility envelope of the gas based on an exploding-wire ignition source and for the apparatus, as described in the previous section.

Figure 6 shows the combustibility range of R134a-air for various initial pressures and molar concentrations. No combustion was observed for concentrations below 10 percent and for pressures below 125 psig. The higher initial pressure tests (400 psig) have the greatest combustibility range with a lower limit of 10 percent and an

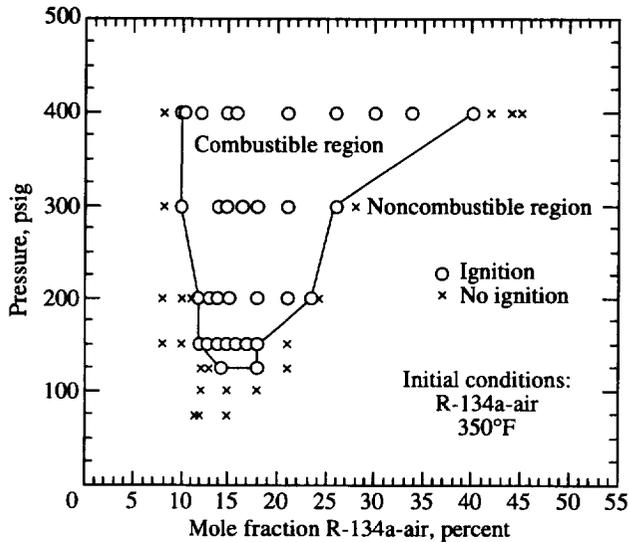


Figure 6. Pressure vs concentration, exploding-wire ignition.

upper limit of 40 percent. The combustibility range for 125-psig initial pressure was shown to be about 14–18 percent. Stoichiometric concentration, as discussed in section 3.4, is 12.28 percent R134a in air.

E. I. DuPont de Nemours Company, a manufacturer of R134a, conducted combustibility tests using the exploding-wire ignitor per ASTM E918-83 (ref. 1) but with a smaller volume test apparatus. The DuPont vessel was a 316 stainless steel pressure vessel with an internal volume of 1.7 L. The criterion for combustion was a pressure increase of 7 percent. DuPont tested a variety of concentrations and initial pressures of <20, 20, 162, and 308 psig. Initial temperatures were 20°C (68°F), 100°C (212°F), and 177°C (350°F). The 177°C tests showed the widest range in the combustibility envelope with minimum molar concentration of R134a-air of 6.5 percent at a pressure of 20 psig. The rich limit at 162 psig was shown to be 19.5 percent R134a. The lowest initial pressure at which combustion occurred was 5 psig. Further increase in initial pressure widened the combustibility envelope such that, at 308 psig, the rich limit was 36.5 percent. The lower initial pressure at which combustion occurred for the DuPont data, as compared to the current study, was attributed to the smaller volume of the test chamber. Given an equal amount of combustible material consumed in the two test chambers, the smaller volume would record the greater pressure increase.

Figures 7 and 8 show initial-to-final-pressure ratio versus concentration for the 300-psig and 400-psig-initial-pressure cases. For the two cases, the pressure peak occurs at the stoichiometric (12.3-percent molar concentration) to slightly rich molar concentration and has a ratio of final-to-initial-pressure ratio of about 5.2:1.

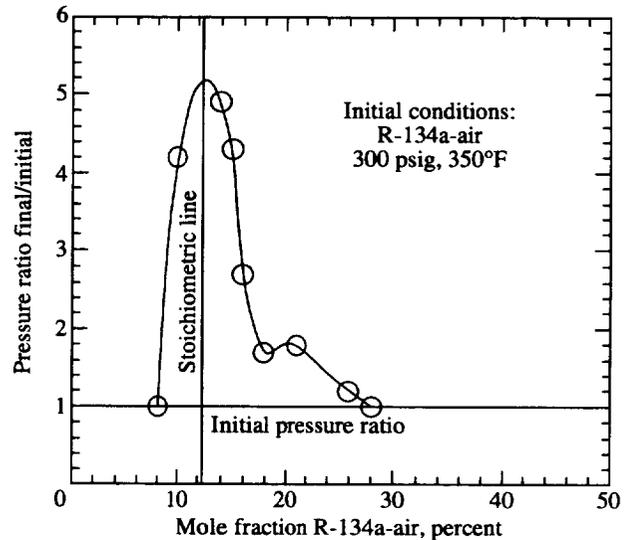


Figure 7. Pressure ratio vs concentration, exploding-wire ignition.

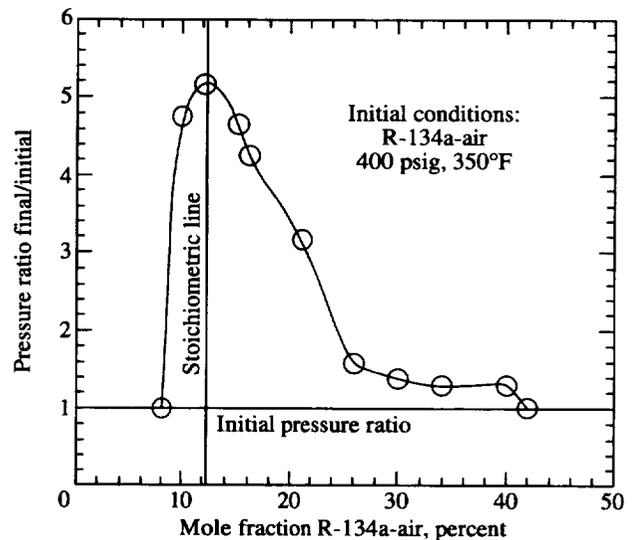


Figure 8. Pressure ratio vs concentration, exploding-wire ignition.

The ratio decreased for initial pressures below about 200 psig, but the peak remained at about the stoichiometric point. The DuPont data indicate a peak pressure of five times the initial pressure that occurred at a molar concentration of 13.5 percent. It appears that a final-to-initial-pressure ratio of approximately 5 is the maximum obtainable and occurs with a concentration at or near stoichiometric. This pressure ratio also indicates that all the O<sub>2</sub>-R134a in the mixture was consumed. For a lower initial pressure stoichiometric mixture, the flame propagation from the ignition source could not sustain itself and was extinguished before all reactants were consumed, resulting in a pressure ratio less than 5.

Imperial Chemical Industries (ICI) conducted combustibility tests in a 490-in<sup>3</sup> (8-L), 6-in.-diameter autoclave. This vessel was similar in size to that used in the current study. Tests were conducted at temperatures up to 170°C (338°F) with a range of molar concentrations and initial pressures. Combustion only occurred for pressures above 50 psia, which is less than the minimum pressure of 125 psia for combustion observed in the current study. The difference is attributed to the ignition source used in the two tests. ICI used a hot-glowing-wire source that consists of passing a current of 4–10 A through a wire for tens of seconds. The hot-glowing-wire ignition source provides sufficient energy to raise local temperatures into the range of autoignition and, therefore, tends to widen the combustibility envelope.

Figure 9 shows a pressure trace for an exploding wire test with a near stoichiometric mixture and initial pressure of 400 psig. As shown in the figure, the peak pressure is about five times the initial pressure. The pressure line shows three distinct slopes. There is an initial slope as the flame propagates from the exploding-wire source. During this initial phase, a polytropic increase in total pressure and temperature in the unburned region occurs. There appears to be a transition or moderation perhaps as the fireball encounters the nearest wall. At this point, the unburned gases are near autoignition conditions, as described in section 3.4. Further propagation of the flame results in an extremely fast rate of pressure rise, of about 3500 psi/sec. This rate is typical

of energetic ignitions. Lower energy ignitions, such as very fuel-rich or low-initial-pressure ignitions, typically had a single slope trace as either the flame quenched or the reactants did not autoignite. The initial slope of the pressure curve of figure 9 is about 1000–1200 psi/sec. This result is near the value of the 1500-psi/sec peak reported by DuPont for all tests. Hoechst measured pressure derivatives from 1400–2500 psi/sec. These values are similar to the initial and peak derivatives measured in the current study. It appears that the closer the mixture is to autoignition conditions at the time of ignition the greater the rate of pressure increase.

**3.1.2 R134a-Air-N<sub>2</sub>.** Table 2(b) summarizes the R134a-air-N<sub>2</sub> tests. Listed are the run number, percent R134a by mole, percent O<sub>2</sub> by mole in the O<sub>2</sub>-N<sub>2</sub> system, pressures of R134a, N<sub>2</sub>, air, equivalence ratio, peak pressure, rise ratio, and final temperature (T1 and T2). Tests were conducted at 200, 300, 400, and 600 psig initial pressure. For the 200- and 300-psig cases, very little nitrogen diluent was needed to render the mixture noncombustible. It was found that, for any mixture in which the O<sub>2</sub> was less than 17 percent of the oxygen-lean air, the mixture was noncombustible. As would be expected with increasing initial pressure, a greater amount of N<sub>2</sub> was required to render the mixture noncombustible. For a 600-psig initial pressure, it would be necessary to double the nitrogen content of the air to preclude combustion. Figure 10 shows the lower limits of

Dataset name	TEST82_CH2	Sample rate	200 data points/sec
Version number	1	Maximum	2153 psig
Series name	Series_1	Minimum	399 psig
Date acquired	9-07-94	Comments	12 percent R134a
Time acquired	17:03:52.40		18.5 percent oxygen
Number samples	2586		

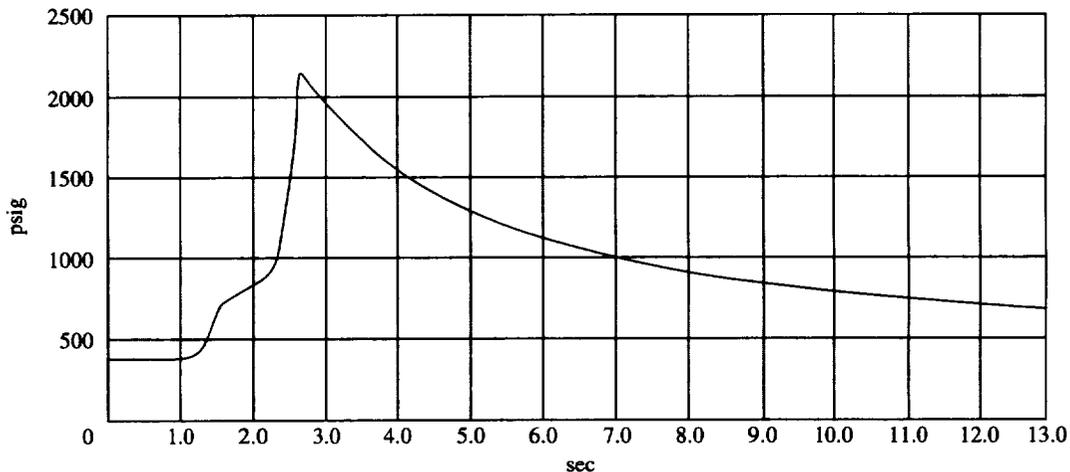


Figure 9. Series 1, test 82, channel 2.

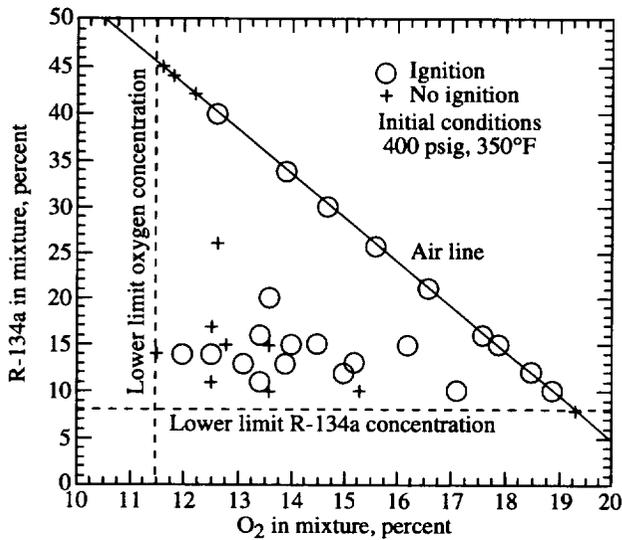


Figure 10. Combustible concentration limits of R134a-O<sub>2</sub> mixtures with exploding wire ignition.

fuel and oxygen molar concentration for the 400-psig-initial-pressure, exploding-wire case with nitrogen added.

ICI conducted studies on the effects of oxygen-deficient air on the combustion of R134a-air. Their work showed that a mixture was combustible with as little as 10 percent O<sub>2</sub> in the total mixture for a 300-psig initial pressure. As discussed previously, the ICI data were generated with a hot-glowing-wire ignition source. This ignitor raises temperatures locally to the autoignition temperature thereby expanding the combustibility envelope.

### 3.2 Cartridge-Heater Tests

Table 2(c) contains a tabulation of results from cartridge-heater tests. Included in the results are the run number, a note on the particular test, molar concentration, R134a gauge pressure, final gauge pressure, equivalence ratio, peak pressure, ratio of initial-to-final pressure, and upper and lower peak thermocouple readings. The cartridge heater, described in section 2.2, protrudes about midway into the chamber interior. The last inch is heated with an energy output of 150 W. An internal thermocouple monitors the cartridge-heater temperature. The cartridge heater simulates a hot spot on an interior surface.

The original intent of the cartridge-heater tests was to simulate a hot wear surface in the compressor. Tests were conducted in the short apparatus with an initial temperature of the gaseous mixture of 350°F. Typically, the tests consisted of ramping the cartridge-heater surface to a temperature of 1500°F in 2 min and then holding the

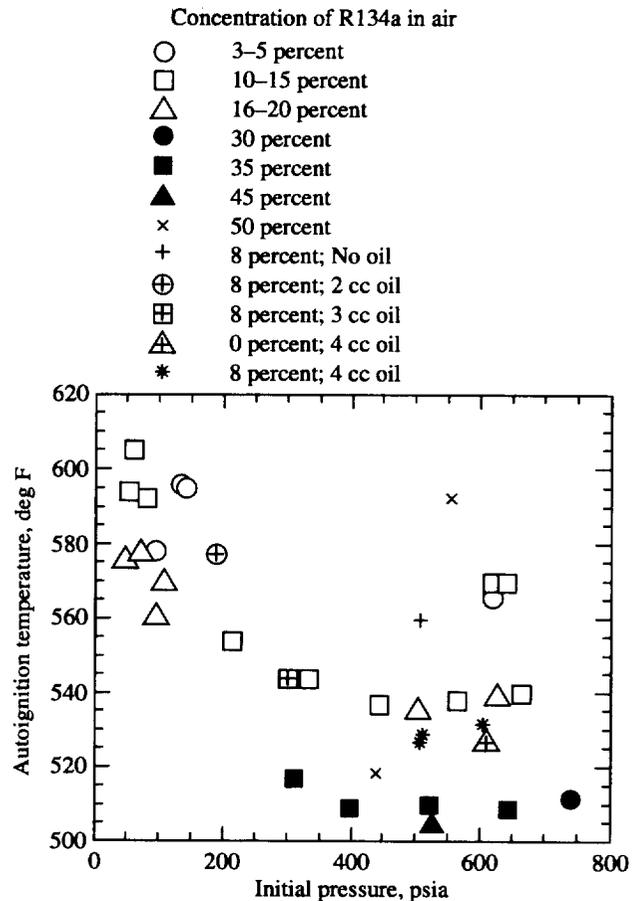


Figure 11. Autoignition temperature vs pressure, R134a-air.

temperature for 2–3 min. Because of the low thermal mass and relative insulating properties of the surrounding gas, the cartridge-heater temperature very closely followed the command temperature during these tests. This temperature was monitored but not recorded. If the mixture failed to ignite after the high-temperature soak, the exploding wire was energized. In the two cases in which the mixture did not ignite with the cartridge heater, it also failed to ignite with the exploding wire. In one case, run 9, ignition of the mixture was attempted first with the exploding wire; then the cartridge heater was energized. In this case, the exploding wire failed to ignite the mixture; however, the cartridge heater ignited the mixture. It was typical in these tests for the mixture to ignite as the temperature of the heater climbed. Ignition occurred in the range of 1000–1500°F. It is thought that the mechanism of ignition is the rising of the local ignition temperature to autoignition. From that point, the flame propagates into the unburned region. This type of ignition is similar to that used by ICI in their tests. Such ignition methods expand the observed combustibility envelope relative to results obtained by the exploding-wire method.

### 3.3 Flame Propagation Through an Obstruction

An investigation was conducted to characterize flame propagation through an obstruction and into a downstream piping system, as might occur in the compressor-stage discharge piping. It was believed that a flame originating in a cylinder of the compressor might be quenched as it passed through the labyrinth path of a discharge valve. Section 2 describes the vessel, piping extension, and discharge valve. Table 3 is a tabulation of the results that lists R134a molar concentration, initial pressure, zone temperature, and propagation status of the test, which were determined by observing the pipe thermocouples. In figure 2, zone 1 contains the short apparatus except for the flanges that hold the valve. Zone 2 contains the flanges, valve, and pipe extension. Each zone was temperature controlled separately. The results shown were all conducted at 300 psig initial pressure, 15-percent R134a molar concentration, and a zone 1 temperature of 350°F. Zone 2 temperature varied from 250–320°F. Ignition was provided by the exploding wire.

The results of these tests showed that valve and piping temperatures of 315°F or less quenched a flame passing through the discharge valve, while a temperature of 320°F allowed the propagation of the flame into the downstream pipe.

It should be noted that dynamic conditions exist in the compressor system; however, the test was initiated with the mixture in a quiescent state. Expansion of the gas caused by heating provided the motivation for flow through the discharge valve. In the actual compressor system, the gas is forced through the discharge valve by the piston. Thus, the tests did not reproduce compressor operating conditions, but do indicate that propagation is possible if a flame is initiated in the cylinder and the discharge valve is operating at nominal temperatures.

### 3.4 Autoignition Tests

Autoignition tests were conducted in the short-test apparatus, as described in section 2. The vessel was initially charged with the desired mixture of R134a and air. The surface heaters were then ramped at about 5–8°F/min until ignition occurred. It was assumed that ignition initiates on a vessel surface; however, the higher reading thermocouple in the gas cavity was taken as the autoignition temperature. The actual temperature of the initiation site was unknown but was estimated to be 5–10°F greater than the bulk gas temperature. The initial pressure was taken as the pressure at the time of ignition, as opposed to the charge pressure. Typically, tests were repeated for calibration and repeatability when a change in a parameter, such as concentration, pressure, or oil contamination was made. Autoignition temperature repeatability was about 6°F, as indicated by the cavity

thermocouples. Tables 4(a), (b), and (c) and table 5 show results of the autoignition tests.

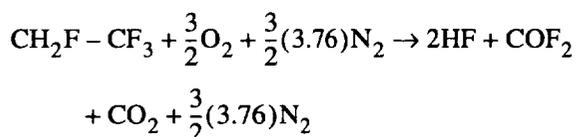
Figure 11 shows the autoignition temperature versus pressure for various mixture concentrations. The curve shows a decreasing autoignition temperature with increasing pressure, with a minimum ignition temperature occurring at about 500-psia initial pressure. One test was performed at atmospheric pressure but failed to ignite at a temperature of 610°F, the maximum temperature allowed for the apparatus. DuPont lists atmospheric pressure autoignition temperature at 1418°F. Hoechst, in their investigation, found atmospheric autoignition occurring at 1100°F and an autoignition temperature of 734°F for an initial pressure of 43.5 psia. For the current study, it was found that the autoignition temperature decreased with increasing R134a mole fraction to a minimum of about 506°F at 45-percent R134a concentration and 520 psia.

Several tests were performed with Tribol 890 compressor lube oil injected into the apparatus before charging. All tests were conducted with an 8-percent R134a 92-percent air mixture on a molar basis. The 8-percent R134a molar concentration was chosen to ensure adequate free oxygen available for reaction with the oil. Tests were conducted at about 200, 300, 500, and 600 psia. The 500- and 600-psia tests were conducted with 4-cc oil injection. The 200- and 300-psia tests were conducted with 2-cc and 3-cc oil injection, respectively. The amount of oil injected for these tests was calculated to be near the stoichiometric value for the air partial pressure. After each test, an 8-percent R134a run with no oil was conducted to burn out any residual materials. One test at 600 psia was conducted with the oil and no R134a. Results of the oil tests are tabulated in table 5 and shown in figure 11. As was the case without oil additive, the autoignition temperature decreased with increasing pressure. The test with 4-cc oil and no R134a is an indication of autoignition of the oil at 600 psia. The tests with 8-percent R134a and oil show lower autoignition temperatures, which indicates that the initial reaction may be caused by the spontaneous ignition of the oil in the oil-R134a-air mixture. The minimum autoignition temperature for the oil tests was 527°F.

### 3.5 Decomposition Tests

Decomposition tests were conducted to determine the effects of long periods of soak at high temperatures in the presence of pure R134a, R134a-air, and R134a-nitrogen samples. After soak, samples were examined with either a paper test to detect hydrogen fluoride (HF) (a combustion product) or Fourier transform infrared (FTIR) spectroscopy for the detection of additional species, as compared to the control sample. Table 6

summarizes these tests. Results indicate that, at temperatures up to 500°F, there is no decomposition into intermediate species and, therefore, no degradation of gas mixtures at these temperatures. This lack of degradation is an important consideration for use in the TDT. A test conducted with an 18-percent R134a, 82-percent air molar concentration was heated until ignition occurred. HF, CO<sub>2</sub>, COF<sub>2</sub>, and unreacted R134a were detected in the products with a mole fraction of COF<sub>2</sub> of about 20 percent molar. A proposed chemical equation for combustion would be



It was estimated that for a rich mixture of 18-percent R134a (stoichiometric mole percent is 12.28 percent) there would be about 22 percent COF<sub>2</sub> in the products, neglecting the nitrogen that is not detected in the FTIR spectroscopy. This result agrees closely with the measured value, which indicates that the proposed chemical equation is reasonable for predicting final products. The HF in the products with water may form hydrofluoric acid, which is corrosive to metals typically found in a piping system.

Although no additional species were noted, it was suspected that less stable intermediate species were formed that decomposed to form the final products. Trifluoroethylene (CF<sub>2</sub> = CHF) is one such decomposition product that has a combustibility range similar to CH<sub>2</sub>F - CF<sub>3</sub> (R134a). By analogy with tetrafluoroethylene (CF<sub>2</sub> = CF<sub>2</sub>) and vinylidene fluoride (CF<sub>2</sub> - CH<sub>2</sub>), the trifluoroethylene would be expected to be highly combustible even at ambient pressures and temperatures.

#### 4.0 Concluding Remarks

Combustibility tests of R134a-air-N<sub>2</sub> were conducted with an American Society for Testing and Materials (ASTM) standard exploding-wire ignition source in a carbon steel vessel that simulated an upper stage of a large multistage compressor. The tests established the combustibility envelope of the mixtures at various pressures and mole fractions. The minimum pressure

required for combustion with the exploding-wire-ignition source was found to be 125 psig, while a minimum concentration of 10-percent R134a was required for ignition. The maximum concentration in which ignition was observed was found to be 40 percent R134a at 400 psig (maximum test pressure). The envelope, shown in figure 6, represents conditions to be avoided when handling mixtures of R134a-air in the presence of a potential spark-ignition source. Note that, for these types of tests, the vessel can have a significant impact on results. The vessel wall material either can be favorable to burning by supplying free radicals to the reaction thus enhancing the burning rate or retard the reaction by lowering the flame temperature (quenching) or by promoting radical recombinations.

The flame-propagation tests established the minimum pipe surface temperature that would propagate a flame. These results are, again, specific to the test apparatus. The discharge valve through which the flame had to propagate has a large surface-area-to-flow-length ratio, which is a desirable characteristic in a flame arrester. It was concluded that extensive testing should be performed on any device used to prevent flame propagation via quenching in a mixture of R134a-air.

The test results of figure 11 show the influence of initial pressure and concentration on the autoignition temperature. These results were obtained in a carbon steel vessel by heating the exterior of the vessel with one control thermocouple that was surface mounted at the vessel center. It was assumed that ignition of the mixture would initiate at a surface that might be 10°F higher than the bulk gas temperature reported. These results indicate care must be taken when subjecting R134a-air mixtures to heat or compression.

NASA Langley Research Center  
Hampton, VA 23681-0001  
January 7, 1997

#### 5.0 Reference

1. Standard Practice for Determining Limits of Flammability of Chemicals at Elevated Temperature and Pressure. *Annual Book of ASTM Standards*, Vol. 14.02, ASTM-E 918-83, 1983.

Table 1. Physical Properties of 1,1,1,2-tetrafluoroethane  
 [Refrigerant R134a, CH<sub>2</sub>F-CF<sub>3</sub>]

Physical property	R134a	Units
Molecular weight	102.03	lbm/lbmole
Boiling point at 1 atm	-14.9	°F
Freezing point	-149.8	°F
Critical temperature	213.9	°F
Critical pressure	588.9	psia
Density, saturated vapor at boiling point	0.328	lbm/ft <sup>3</sup>
Specific heat, constant pressure at 77°F, 1 atm	0.204	Btu/lbm°F
Vapor pressure at 77°F	96.6	psia
Thermal conductivity vapor, 1 atm	0.00836	Btu/hr-ft°F
Speed of sound	539	ft/sec
Ozone depletion potential (R12 = 1.0)	0	—
Halocarbon global warming potential (HGWP) (R12 = 3.2)	.26	—

Table 2. Test Results of R134a for Revision 5 on April 11, 1995

(a) R134a-air, 350°F initial temperature

[Exploding-wire ignition]

Run	R134a in mix, percent	R134a pressure, psig	Air pressure, psig	${}^a E_q$	Pressure peak, psig	Pressure ratio	${}^c T_1$ , deg F	${}^c T_2$ , deg F
139	10	62.0	600	0.80	3002	4.9	>1652	1436
21	8	33.2	400	0.62	b	b	358	348
20	10	41.5	400	0.79	1644	4.0	1251	1190
69	10	41.5	400	0.79	1971	4.8	1301	1220
82	12	49.8	400	0.98	2153	5.2	1397	1300
83	15	62.2	400	1.26	1950	4.7	1267	1185
81	16	66.4	400	1.36	1782	4.3	1249	1164
24	21	87.2	400	1.90	1298	3.2	1217	1116
68	25.8	106.8	400	2.48	655	1.6	769	376
86	30	124.5	400	3.06	559	1.4	513	355
87	33.8	140.3	400	3.65	508	1.3	466	359
126	40	165.9	400	4.76	506	1.3	419	356
129	42	174.2	400	5.17	b	b	350	350
128	44	182.5	400	5.61	b	b	350	350
127	45	186.6	400	5.84	b	b	350	350
19	8	25.2	300	0.62	b	b	350	350
18	10	31.5	300	0.79	1299	4.2	1149	1075
99	14	44.1	300	1.16	1530	4.9	1116	1121
78	15	47.3	300	1.26	1345	4.3	1134	1124
37	16.4	51.5	300	1.40	847	2.7	1090	1098
100	18	56.6	300	1.57	533	1.7	801	527
23	21	66.2	300	1.90	552	1.8	713	381
67	26	81.8	300	2.51	371	1.2	363	349
66	28	88.0	300	2.78	b	b	350	350
16	8	17.2	200	0.62	b	b	350	350
17	10	21.5	200	0.79	b	b	350	350
26	10	21.5	200	0.79	b	b	350	350
36	11	23.6	200	0.88	b	b	350	350
3	12	25.8	200	0.98	650	3.1	b	b
44	12	25.8	200	0.98	773	3.7	1060	1022
33	13	27.9	200	1.07	788	3.7	1048	994
34	14	30.1	200	1.16	868	4.1	975	960
35	15	32.2	200	1.26	529	2.5	1011	940
103	15	32.2	200	1.26	691	3.3	1001	972
97	18	38.6	200	1.57	891	4.2	923	910
22	21	45.2	200	1.90	260	1.3	365	350
65	24	51.5	200	2.25	232	1.2	361	350
102	24	51.5	200	2.25	b	b	350	350
<sup>d</sup> 104	24	51.5	200	2.25	b	b	350	350
101	24.2	52.0	200	2.28	b	b	350	350
14	8	13.2	150	0.62	b	b	350	350
15	10	16.5	150	0.80	b	b	350	350
10	12	19.9	150	0.98	432	2.7	999	850

All footnotes are on page 12.

Table 2. Continued

(a) Concluded

Run	R134a in mix, percent	R134a pressure, psig	Air pressure, psig	<sup>a</sup> $E_q$	Pressure peak, psig	Pressure ratio	<sup>c</sup> T1, deg F	<sup>c</sup> T2, deg F
25	13	21.5	150.1	1.07	736	4.6	980	858
38	13	21.4	150	1.07	532	3.3	898	907
31	14	23.1	150	1.17	279	1.8	722	558
51	14	23.1	150	1.17	488	3.1	985	908
52	14	23.1	150	1.17	474	3.0	945	901
28	15	24.7	150	1.26	601	3.7	921	868
32	15	24.7	150	1.26	480	3.0	892	895
53	15	24.7	150	1.26	327	2.1	750	758
105	15	24.7	150	1.26	320	2.0	805	691
55	15	24.7	150	1.26	315	2.0	780	602
54	15	24.7	150	1.26	249	1.6	574	360
58	15	24.7	150	1.26	251	1.6	480	359
<sup>e</sup> 56	15	24.7	150	1.26	b	b	340	340
29	16	26.4	150	1.36	280	1.8	648	379
30	17	28.0	150	1.46	284	1.8	596	382
95	18	29.6	150	1.56	262	1.7	550	364
59	18	29.6	150	1.56	264	1.7	523	356
13	21	34.7	150	1.91	198	1.3	361	354
12	12	16.9	125	0.98	b	b	350	350
98	13	18.3	125	1.08	b	b	350	350
61	14.3	20.0	125	1.19	194	1.5	519	356
60	18	25.1	125	1.56	150	1.2	361	349
64	21	29.3	125	1.90	b	b	350	350
<sup>f</sup> 1	11.5	10.7	78.4	0.93	b	b	350	350
6	12	10.8	75.3	0.97	b	b	350	350
<sup>g</sup> 91	12	10.8	75.3	0.97	b	b	350	350
9	15	13.5	75.2	1.26	b	b	350	350
11	12	13.8	100	0.98	b	b	350	350
62	15	17.2	100.1	1.26	b	b	350	350
63	18	20.6	100	1.56	b	b	350	350

<sup>a</sup>Equivalence ratio, fuel lean  $E_q < 1 <$  fuel rich  $E_q \equiv \frac{\text{moles R134a}}{\text{moles O}_2} / \text{stoichiometric ratio}$ .

<sup>b</sup>No combustion observed.

<sup>c</sup>T1 and T2 signify thermocouple 1 and thermocouple 2, respectively.

<sup>d</sup>Mixture soaked 20 min before ignition was attempted.

<sup>e</sup>Vessel temperature low, no ignition.

<sup>f</sup>Failed to ignite with cartridge heater at 1500°F and exploding wire.

<sup>g</sup>Failed to ignite with cartridge heater at 1100°F and exploding wire.

Table 2. Continued

(b) R134a-Nitrogen-air, 350°F initial temperature

[Exploding-wire ignition]

Run	R134a in mix, percent	$\frac{O_2}{O_2 + N_2}$ , percent	R134a pressure, psig	N2 pressure, psig	Air pressure, psig	$^a E_q$	Pressure peak, psig	Pressure ratio	$^c T1$ , deg F	$^c T2$ , deg F
49	10	15.1	41.6	147.1	400	1.11	b	b	350	350
84	10	17	41.5	112.6	400	0.98	b	b	350	350
50	10	19	41.5	77.0	400	0.88	1358	3.3	1360	1217
120	11	14	45.6	168.6	400	1.32	b	b	350	350
119	11	15	45.6	151.1	400	1.24	556	1.4	396	357
111	12	17	49.8	119.3	400	1.20	678	1.7	853	408
114	13	15	53.9	157.0	400	1.49	483	1.2	371	347
113	13	16	53.9	139.8	400	1.40	632	1.6	669	365
110	13	17.5	53.9	114.0	400	1.28	1805	4.4	1138	1070
116	14	13.4	58.1	187.0	400	1.82	b	b	350	350
118	14	14	58.1	177.0	400	1.75	531	1.3	377	354
117	14	14.5	58.1	168.5	400	1.69	589	1.5	490	359
71	15	15	62.2	162.9	400	1.76	b	b	350	350
89	15	16	62.2	146.1	400	1.65	b	b	350	350
112	15	16.5	62.2	137.7	400	1.60	576	1.4	448	356
85	15	17	62.2	129.3	400	1.56	634	1.6	703	366
72	15	19	62.2	95.8	400	1.39	743	1.8	913	600
115	16	16	66.4	149.3	400	1.79	620	1.5	656	361
121	17	15	70.5	168.8	400	2.05	b	b	350	350
122	20	17	82.9	146.1	400	2.20	634	1.6	673	369
88	26	17	107.8	166.3	400	3.10	b	b	350	350
<sup>d</sup> 90	15	12.3	92.2	600.0	<sup>b</sup> 383.4	2.15	775	1.3	374	358
70	9.9	9.9	62.2	361.8	614.6	1.66	b	b	350	350
41	12	10	25.8	124.8	200	2.05	b	b	350	350
42	12	15	25.8	79.8	200	1.37	b	b	350	350
<sup>d</sup> 45	12	16.6	25.8	200.0	<sup>d</sup> 160	1.24	b	b	350	350
43	12	19.4	25.8	40.1	200	1.06	b	b	350	350
46	12	20.3	25.8	32.0	200	1.01	670	3.2	1052	991
125	13	18	27.9	54.6	200	1.24	b	b	350	350
124	13	18.5	27.9	50.1	200	1.21	300	1.5	533	356
123	14	19	30.1	47.7	200	1.29	374	1.8	716	436
106	15	18	32.2	58.3	200	1.47	b	b	350	350
40	8.2	13.8	25.8	124.8	300	0.97	b	b	350	350
47	12	15	38.0	116.9	300	1.37	b	b	350	350
48	12	19	37.8	64.2	300	1.08	b	b	350	350
76	15	17	47.2	98.2	300	1.56	b	b	350	350
75	15	18	47.2	85.4	300	1.47	402	1.3	375	355
74	15	19	47.2	72.3	300	1.39	473	1.5	615	365
77	17	17	53.5	103.3	300	1.81	358	1.2	365	352
79	17	16	53.5	115.7	300	1.92	b	b	350	350
73	19.8	15	62.2	134.8	300	2.47	b	b	350	350

<sup>a</sup>Equivalence ratio, fuel lean  $< E_q = 1 <$  fuel rich  $E_q = \frac{\text{moles R134a}}{\text{moles O}_2} / \text{stoichiometric ratio}$ .<sup>b</sup>No combustion observed.<sup>c</sup>T1 and T2 signify thermocouple 1 and thermocouple 2, respectively.<sup>d</sup>Added N<sub>2</sub> before air.

Table 2. Concluded  
(c) R134a-air, 350°F initial temperature  
[Cartridge-heat data]

Run	Note	R134a in mix, percent	R134a pressure, psig	Air pressure, psig	<sup>a</sup> $E_q$	Pressure peak, psig	Pressure ratio	<sup>c</sup> T1, deg F	<sup>c</sup> T2, deg F
7	1	12	10.8	75.3	0.97	130	1.6	539	417
8	2	15	13.5	75.3	1.26	139.8	1.7	537	417
9	3	15	13.5	75.2	1.26	164	2.0	583	474
92	8	18	20.9	100	1.59	b	b	350	350
2	4	12	19.8	150	0.98	b	b	350	350
5	5	12	19.8	150	0.98	525	3.3	967	836
4	6	12	22.8	175	0.97	800	4.3	948	885
1a	7	12	25.8	200	0.97	860	4.1	980	870

<sup>a</sup>Equivalence ratio, fuel lean  $E_q < 1 <$  fuel rich  $E_q \equiv \frac{\text{moles R134a}}{\text{moles O}_2} / \text{stoichiometric ratio}$ .

<sup>b</sup>No combustion observed.

<sup>c</sup>T1 and T2 signify thermocouple 1 and thermocouple 2, respectively.

Notes: Ramped to 1500°F in 2 min, held for 3 min

1. Ignition with cartridge heater ~1500°F
2. Ignition with cartridge heater ~1400°F
3. Failed to ignite with exploding wire, ignited with cartridge heater ~1400°F
4. Failed to ignite with cartridge heater at 1500°F and exploding wire ignitor
5. Ignition with cartridge heater ~1080°F
6. Ignition with cartridge heater ~1100°F
7. Ignition with cartridge heater ~1480°F
8. Failed to ignite with cartridge heater at 1100°F and exploding wire ignitor (ramped to 1100°F in 2 min, held for 3 min)

Table 3. Test Results R134a-air for Revision 1 on April 11, 1995

[Propagation studies with exploding-wire ignition]

Run	R134a in mix, percent	Initial pressure, psig	Zone 1 set temperature, deg F	Zone 2 set temperature, deg F	Comments
2-26	15	300	350	250	Ignition in zone 1, no propagation to zone 2
2-27	15	300	350	275	Ignition in zone 1, no propagation to zone 2
2-28	15	300	350	300	Ignition in zone 1, no propagation to zone 2
2-29	15	300	350	310	Ignition in zone 1, no propagation to zone 2
2-30	15	300	350	320	Ignition in zone 1, propagation to zone 2
2-31	15	300	350	315	Ignition in zone 1, no propagation to zone 2

Note: Stoichiometric concentration = 12.3 percent.

Table 4. Test Results R134a for Revision 2 on April 11, 1995

(a) R134a-air selected data runs between 93 and 135

[Autoignition]

Run	R134a in mix, percent	R134a pressure, psig	Air pressure, psig	Notations
93	Unknown	13.5	109	600°F initial temperature. From initial charge of air at atmospheric pressure, spike noted when R134a added. Final pressure of 13.5 psig noted: attempted to add air to 75.3 psig. Spike of 109.3 psig noted after charging. Mixture did not ignite with cartridge heater.
94	41	16.2	57.3	600°F initial temperature. From initial charge of air at atmospheric pressure, 10 psig R134a introduced. After about 1.5 min, pressure climbed to 15.5 psig. Added 16.2 psig R134a. Introduced air to 56 psig: instant rise to 88 psig and temperature rise to 732°F and 703°F on top and bottom.
94(a)	18	16.2	75.3	500°F initial temperature. Ignited with cartridge heater: 638°F temperature peak. Spontaneous pressure rise not noted for this case.
94(b)	52	16.2	75.3	550°F initial temperature. From initial charge of 16.2 psig R134a, pressure jumped to 20.5 psig in about 1 min. Charged with air to 70 psig: pressure spiked immediately to 125 psig.
109	18	16.2	75.3	Charged vessel with R134a and air in proportions indicated. 350°F initial temperature. Set controller to 600°F; temperature ramped. 26 min into test, pressure rise from 93 psig to 235 psig noted (instantaneous rise). Temperature rise from 57°F to 780°F noted.
130	18	16.2	75.3	350°F initial temperature; took sample. Ramped to 550°F; took sample. Ramped to 600°F; spontaneous ignition: pressure rose from 59 psig to 92 psig. At T1, temperature rose from 578°F to 707°F. At T2, temperature rose from 570°F to 673°F; 1.5X pressure rise (02/04/94).
131	<sup>a</sup> 18	16.2	75.3	350°F initial temperature. Ramped to 600°F; spontaneous ignition: Pressure rose from 82 psig to 130 psig, 1.5X pressure rise. Temperature T1 rose from 561°F to 697.2°F; temperature T2 rose from 567°F to 683°F (02/24/94).
132	5	4.5	75.3	350°F initial temperature. Ramped to 600°F; spontaneous ignition: pressure rose from 80.1 psig to 108 psig, 1.3X pressure rise. Temperature T1 rose from 578°F to 648°F; temperature T2 rose from 576°F to 620°F. (02/24/94, low-concentration test)
133	2	1.8	75.3	350°F initial temperature. Ramped to 600°F; no ignition. (02/24/94, low-concentration test)
134	20	4.0	5.0	350°F initial temperature. Ramped to 600°F; no ignition. (02/25/94, low-pressure test)
135	20	18.0	75.3	After charge, reduced pressure to 25 psig. 350°F initial temperature. Ramped to 600°F; pressure rose from 32 psig to 53 psig. Temperature T1 rose from 576°F to 676°F; Temperature T2 rose from 582°F to 669°F, 1.5X pressure rise. (02/25/94, low-pressure test, spontaneous ignition)

<sup>a</sup>11.5 percent O<sub>2</sub> and 70.5 percent N<sub>2</sub>.

Table 4. Continued

(b) R134a-air selected data runs between 136 and 144 and between 2-11 and 2-35

[Autoignition]

Run	R134a in mix, percent	Ignition pressure, psig	Pressure peak, psig	Pressure ratio	<sup>c</sup> T1, deg F	<sup>d</sup> T2, deg F	Temperature status	Ignition
136	16	612	2571	4.1	540 1592	473 1229	Initial Final	Spontaneous
137	10	625	2475	3.9	570 1565	506 1282	Initial Final	Spontaneous
140	10	605	2728	4.4	570 >1652	567 1436	Initial Final	Spontaneous
141	12	649	3127	4.7	540 >1652	538 1629	Initial Final	Spontaneous
142	5	605	1446	2.4	566 1018	570 997	Initial Final	Spontaneous
144	18	490	2375	4.7	536 1399	535 1230	Initial Final	Spontaneous
144a	15	430	1737	3.9	537 1425	536 1425	Initial Final	Spontaneous
144b	15	550	2063	3.7	539 1526	539 1364	Initial Final	Spontaneous
2-11	15	319	a		544 949	b	Initial Final	Spontaneous
2-12	15	199	c		544 880	d	Initial Final	Spontaneous
2-25c	15	44	c		605 692	d	Initial Final	Spontaneous
2-32	15	67	c		592 716	d	Initial Final	Spontaneous
2-33	10	38	c		594 706	d	Initial Final	Spontaneous
2-34	5	120	c		596 700	d	Initial Final	Spontaneous
2-35	3	128	c		595 671	d	Initial Final	Spontaneous

Note: All footnotes are located on page 18.

Table 4. Concluded  
(c) R134a-air selected data runs between 145 and 159  
[Autoignition]

Run	R134a in mix, percent	Ignition pressure, psig	Pressure peak, psig	Pressure ratio	<sup>c</sup> T1, deg F	<sup>d</sup> T2, deg F	Temperature status	Ignition
145	30	727	2282	3.1	512 1194	512 1124	Initial Final	Spontaneous
146	35	630	1116	1.8	507 878	509 857	Initial Final	Spontaneous
147	35	505	852	1.7	507 798	510 792	Initial Final	Spontaneous
148	35	384	648	1.7	508 765	509 743	Initial Final	Spontaneous
149	35	296	548	1.8	517 800	516 785	Initial Final	Spontaneous
150	45	511	769	1.5	506 707	504 689	Initial Final	Spontaneous
159	50	427	617	1.4	519 689	515 664	Initial Final	Spontaneous

<sup>a</sup>Tests conducted with extended vessel with one end heated to autoignition.

<sup>b</sup>No temperature recorded at T2.

<sup>c</sup>The higher temperature of T1 and T2 is taken as the autoignition temperature.

<sup>d</sup>Series 2 tests conducted with extended vessel.

Table 5. Test Results R134a-Oil for Revision 1 on April 11, 1995

[Autoignition]

Run	R134a in mix, percent	Oil in <sup>a</sup> mix, cc	Ignition pressure, psig	Pressure peak, psig	Pressure ratio	<sup>b</sup> T1, deg F	<sup>b</sup> T2, deg F	Temperature status	Ignition
152	8	0	492	1615	3.2	560 1154	560 1145	Initial Final	Spontaneous
153	8	4	493	1849	3.7	527 1279	523 1268	Initial Final	Spontaneous
153a	0	4	596	695	1.2	527 592	522 588	Initial Final	Spontaneous
154	8	3	285	1008	3.4	544 1039	539 1055	Initial Final	Spontaneous
155	8	2	174	463	2.5	577 809	570 770	Initial Final	Spontaneous
157	8	4	589	2293	3.8	532 1431	528 1315	Initial Final	Spontaneous
158	8	4	499	1904	3.7	529 1355	527 1258	Initial Final	Spontaneous

<sup>a</sup>Mixed with Tribol 890 lubricating oil.

<sup>b</sup>The higher temperature of T1 and T2 is taken as the autoignition temperature.

Table 6. Decomposition Tests

Mixture	Pressure, psig	Temperature, deg F	Soak time, hr
<sup>a</sup> Pure R134a	50	350	24
<sup>a</sup> Pure R134a	50	350	72
<sup>b</sup> Pure R134a	50	350	336
<sup>b</sup> Pure R134a	50	600	168
<sup>a</sup> 18 percent R134a-82 percent air	150	350	24
<sup>c</sup> 18 percent R134a-82 percent air	150	350	24
<sup>b</sup> 18 percent R134a-82 percent air	150	350	24
<sup>b</sup> 18 percent R134a-82 percent air	150	500	24
<sup>d</sup> 18 percent R134a-82 percent air	150	578	—
<sup>b</sup> 20 percent R134a-80 percent N <sub>2</sub>	62	600	1
<sup>b</sup> 20 percent R134a-80 percent air	50	350	24
<sup>b</sup> 20 percent R134a-80 percent air	50	400	24
<sup>b</sup> 20 percent R134a-80 percent air	50	450	48

<sup>a</sup>No HF detected (indicator paper test).

<sup>b</sup>Species identical to control sample (no combustion) FTIR.

<sup>c</sup>Combusted with exploding wire; HF detected (indicator paper test).

<sup>d</sup>Ignition occurred (autoignition) HF, CO<sub>2</sub>, COF<sub>2</sub>, and unreacted R134a detected FTIR.

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